Site-Specific Standard Operating Procedure for Data Validation of Asbestos Results Obtained by Scanning Electron Microscopy for the Contaminant Screening Study of the Libby Asbestos Project

Project Libby Asbestos Remedial Investigation - Contaminant Screening Study (CSS)

Project Number 3282-116

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Approved by _____
Project Manager — Date

_____
Technical Reviewer — Date

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QA Reviewer — Date

Default goals

No U S Environmental Protection Agency (EPA) approved criteria currently exists for the validation of asbestos results The following procedures for data validation are based on the EPA Contract Laboratory Program (CLP) National Functional Guidelines for Inorganic Data Review (EPA 1994) and EPA Standard Operating Procedure (SOP) No EPA-LIBBY-01, Asbestos Analysis of Soils by Scanning Microscopy and Energy Dispersive X-Ray Spectroscopy (EPA 2000) These procedures will be used in the data validation and evaluation process for results gathered as part of the contaminant screening study (CSS) of the Libby Asbestos Project This is a working document and applicable changes will be made as the validation procedure is implemented

Section 1
Calibration Criteria
1 1 Initial Calibration

The scanning electron microscope (SEM) is calibrated with four standards at the following minimum frequency (1) prior to receipt of samples, (2) monthly after first calibration and (3) after any maintenance Data packages will be checked to ensure that the following initial calibration standards are met and performed at the required frequency Initial calibration consists of magnification calibration, peak centroid calibration, resolution calibration, and sodium sensitivity If the laboratory has failed to provide adequate calibration information the designated representative should contact the laboratory and request the necessary information

Evaluation Verify initial calibration was performed at the proper frequency

Action Minimum frequency was not met, qualify the data as unusable (R)

1 1 1 Magnification Calibration

The magnification calibration should fall within ± 10 percent of the certified values as indicated in the calibration standard manufacturer s specifications The results of this calibration are recorded on the data collection logsheet

Evaluation Verify magnification calibration is within ± 10 percent of the certified values

Action

Certified Value	Detected Results Qualifier	Nondetected Results Qualifier
0 to +25%	J	No qualifier
0 to 25%	J	UJ
< 25%	R	~~No qualifier~~
> +25%	R	No qualifier

1 1 2 Peak Centroid Calibration

The aluminum centroid peak should be 1 487 (± 0 05) KeV and the copper centroid peak should be 8 047 (± 0 05) KeV The results of this calibration are recorded on the data collection logsheet

Evaluation Verify peak centroid calibration is within ± 0 05 KeV of the certified values

Action

Centroid Calibration	Detected Results Qualifier	Nondetected Results Qualifier
> ± 0 05 KeV but < ± 0 25 KeV	J	UJ
> ± 0 25 KeV	R	R

1 1 3 Resolution Calibration

The resolution must be no greater than 175 eV The results of this calibration are recorded on the data collection logsheet

Evaluation Verify resolution calibration is ≤ 175 eV

Action

Resolution	Detected Results Qualifier	Nondetected Results Qualifier
>175 eV but <200 eV	J	UJ
>200 eV	R	R

OMIT ??

1 1 4 Sodium Sensitivity

The sodium sensitivity calibration should be performed in accordance with the manufacturer s specifications These specifications and the acceptable criteria should be included in each data package

Evaluation ???

Action ???

SAME AS 1R

1 2 Continuing Calibration

An independent laboratory control sample (LCS) must be analyzed with each analytical batch or once a day, whichever is more frequent An analytical batch is comprised of 20 field samples The acceptable percent recovery (%R) for continuing calibration criteria is between 80 and 120%R %R is calculated by the following

$$\%R = \frac{\text{Found}}{\text{True}} \times 100$$

Where Found = result of asbestos (percent weight) measured in the LCS

True = result of asbestos (percent weight) in the LCS source

Evaluation Verify continuing calibration was performed at the required frequency

Action Minimum frequency was not met qualify the data as unusable (R)

Evaluation Verify continuing calibration is between 80 and 120%R

Action

%R	Detected Results Qualifier	Nondetected Results Qualifier
65 79%	J	UJ
121 135%	J	No qualifier
<65%	R	No qualifier
>135%	R	No qualifier

Section 2
Method Blanks

SAME AS IR

An instrument blank is composed of the field sample matrix that is free of the analyte of interest (e g asbestos-free soil) Method blanks are put through the same sample preparation steps as field samples and are used to discern if laboratory-induced contamination is present Detection of a single asbestos fiber suggests that laboratory-induced contamination is present All associated samples may require re-preparation and re-analysis Method blanks must be analyzed with each analytical batch or once a day, whichever is more frequent An analytical batch is comprised of 20 field samples Multiplying the highest concentration of asbestos detected in the method blank times five gives the action level for qualification based on method blank contamination

Evaluation Verify method blank analysis was performed at the required frequency

Action Minimum frequency was not met the validator should use professional judgment to determine if the associated sample results should be qualified

Evaluation Calculate the method blank action level for qualification

Action All detected results less than the action level are qualified as estimated (U)

Section 3
Laboratory Control Sample (LCS)

SAME AS IR

Laboratory control samples are certified reference standards (independent from the calibration standards), consisting of several asbestiforms Because LCSs are independent of the calibration standards, they are analyzed to verify the accuracy of the standards used to calibrate the instrument An LCS must be analyzed with each analytical batch or once a day whichever is more frequent The LCS will be evaluated

on two parameters and it must meet the acceptance criteria for both to be considered acceptable These parameters are (1) accurate asbestiform identification and (2) accurate fiber counting and sizing The acceptable percent recovery (%R) for continuing calibration criteria is between 80 and 120%R %R is calculated by the following

$$\%R = \frac{\text{Found}}{\text{True}} \times 100$$

Where Found = result of asbestos (percent weight) measured in the LCS

True = result of asbestos (percent weight) in the LCS source

Evaluation Verify LCS analysis was performed at the required frequency

Action The validator should use professional judgment to determine if the associated sample results should be qualified

Evaluation Verify LCS result is between 80 and 120%R

Action

%R	Detected Results Qualifier	Nondetected Results Qualifier
65 79%	J	UJ
121 135%	J	No qualifier
<65%	R	No qualifier
>135%	R	No qualifier

Section 4
Duplicate Sample Analysis

4 1 Laboratory Duplicate Samples

SAME AS SA 1R

Laboratory duplicate samples are splits of a well-homogenized sample that is prepared by the laboratory personnel Because the laboratory is aware that the samples are duplicates, these samples serve to test the precision of the laboratory s sample preparation and analysis A laboratory duplicate should be performed at a frequency of 5 percent of all field samples prepared for analysis (one laboratory duplicate for every 20 field samples) or one per preparation batch whichever is more

frequent The acceptable criteria for a laboratory duplicate is a relative percent difference (RPD) less than or equal to 35 percent when both results are >5 times the reporting limit, or the difference between the duplicate and the original is less than two times the reporting limit when either sample result is <5 times the reporting limit

Evaluation Verify laboratory duplicate sample analysis was performed at the required frequency

Action The validator should use professional judgment to determine if the associated sample results should be qualified

Evaluation Verify RPD ≤ 35 percent, or difference is less than two times the reporting limit, whichever is applicable

Action RPD > 35 percent or difference is greater than two times the reporting limit, qualify all results as estimated (J)

SAME SOP AS IR

4 2 Field Duplicate Samples

Field duplicate samples are co-located soil samples that are collected by the field personnel but the laboratory is unaware that the samples are duplicates These samples serve to test the precision of both the field sampling and the laboratory s sample preparation and analysis A field duplicate should be collected at a frequency of 5 percent of all field samples prepared for analysis (one laboratory duplicate for every 20 field samples) or one per preparation batch whichever is more frequent The acceptable criteria for a field duplicate is an RPD less than or equal to 50 percent when both results are >5 times the reporting limit or the difference between the duplicate and the original is less than four times the reporting limit when either sample result is <5 times the reporting limit

Evaluation Verify field duplicate sample analysis was performed at the required frequency

Action The validator should use professional judgment to determine if the associated sample results should be qualified

Evaluation Verify RPD ≤ 50 percent or difference is less than four times the reporting limit whichever is applicable

Prep blank

Action RPD > 50 percent or difference is greater than four times the reporting limit qualify all results as estimated (J)

4 3 Preparation Duplicate Samples

Preparation duplicate samples are splits of samples submitted for sample preparation prior to laboratory analysis These samples serve to test the precision of both the sample preparation personnel and the laboratory s sample preparation and analysis

A preparation duplicate sample should be submitted at a frequency of 5 percent of the first 500 field samples prepared for analysis or one per preparation batch whichever is more frequent The acceptable criteria for a field duplicate is an RPD less than or equal to 50 percent when both results are >5 times the reporting limit or the difference between the duplicate and the original is less than four times the reporting limit when either sample result is <5 times the reporting limit If the average RPD for the first 500 samples is ≤50 percent, preparation duplicate sample analysis will not continue If the average RPD for the first 500 samples is >50 percent preparation duplicate sample analysis will continue at a rate of 2 percent for the remainder of the project

Evaluation Verify preparation duplicate sample analysis was performed at the required frequency

Action The validator should use professional judgment to determine if the associated sample results should be qualified

Evaluation Verify RPD ≤ 50 percent or difference is less than four times the reporting limit whichever is applicable

Action RPD > 50 percent or difference is greater than four times the reporting limit qualify all results as estimated (J)

4 4 IR and SEM Sample Splits

Selected field samples will be analyzed by both infrared spectroscopy (IR) and SEM methods The sample results will be compared to determine if the IR results and SEM results are within an acceptable RPD range The acceptable criteria for a laboratory duplicate is an RPD less than or equal to 35 percent when both results are >5 times the reporting limit or the difference between the duplicate and the original is less than two times the reporting limit when either sample result is <5 times the reporting limit

Evaluation Verify RPD ≤ 35 percent, or difference is less than two times the reporting limit

Action RPD > 35 percent or difference is greater than two times the reporting limit qualify all results as estimated (J)

Section 5
Rinsate Samples

Rinsate samples are collected to determine if decontamination procedures utilized in the field are not adequate and result in cross-contamination of samples Rinsate samples will be collected at the end of each day during the first week of sampling Continuation of rinsate sample collection will depend on the results of the initial rinsate samples Multiplying the highest concentration of asbestos detected in the

rinsate times five gives the action level for qualification based on contamination from sampling equipment

- **Evaluation** Verify rinsate sample analysis was performed at the required frequency
- **Action** Minimum frequency was not met the validator should use professional judgment to determine if the associated sample results should be qualified
- **Evaluation** Calculate rinsate sample action level for qualification
- **Action** All associated detected results less than the action level are qualified as nondetect (U)